

Data Compilation for AGC-2 Matrix-only Compact Lot A3-H08

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Data Compilation for AGC-2 Matrix-only Compact Lot A3-H08

J. D. Hunn and M. P. Trammell Oak Ridge National Laboratory

This document is a compilation of fabrication and characterization data for the compact lot A3-H08, which was produced for possible insertion into the second ATR Graphite Creep irradiation test capsule (AGC-2). The compacts were produced by Oak Ridge National Laboratory (ORNL) for the Advanced Gas Reactor Fuel Development and Qualification (AGR) program. This compact lot was fabricated using a graphite/resin blend formulated by INL as a candidate matrix precursor for scale-up of overcoating and compacting processes. The matrix precursor powder for compact lot A3-H08 was a jet milled blend of 64 wt% natural graphite (Asbury 3482), 16 wt% synthetic graphite (Graftech GTI-D), and 20 wt% high purity novolac resin (Hexion D_SD-1708 with 7.5% hexamethylenetetramine added).

The "Matrix Compacts for AGC-2 Irradiation" Specification (INL SPC-1285, Rev. 1) provides the requirements necessary for acceptance of the compacts. Sections 2.03 and 2.04 of SPC-1285 provide the property requirements for the heat-treated compacts. There are requirements on the length, diameter, and matrix density. Section 2.02.01.01 further requests impurity analysis on representative samples from the final compact lot, but there are no specified acceptance criteria. The impurity information will be evaluated by INL as part of a final decision on whether to insert the compacts into the AGC-2 experiment. The procedures for characterizing and qualifying the compacts are outlined in ORNL product inspection plan AGR-CHAR-PIP-16. The measurement of compact length, diameter, and matrix density was performed according to data acquisition method AGR-CHAR-DAM-39. The data report forms generated by this procedure document the product acceptance for the property requirements listed in sections 2.03 and 2.04 of SPC-1285. All compacts selected for irradiation conformed to the specified requirements for length, diameter, and density.

In addition to the characterization data, this report also contains other records relevant to the product acceptance. A history of the material flow and sample naming is included and the compacting process is summarized. In addition to results of the impurity analysis on representative samples from the final compact lot, impurity analysis results are also provided for samples of the graphite, resin, and the graphite/resin blend. A Certificate of Conformance to SPC-1285, Rev. 1 is attached in Appendix B.

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1 Material identification record for A3-H08

Table 1-1 lists the materials used to make the A3-H08 compacts. The graphite/resin blend was shipped from Babcock and Wilcox (B&W) to ORNL on October 4, 2010. Three completed compacts were shipped to INL on October 28, 2010. Three compacts were also shipped to Shiva Technologies for impurity analysis on October 29, 2010. Fourteen compacts were retained at ORNL. Table 1-2 lists the disposition of each compact. Table 1-3 is a copy of data report form DRF-39B, which lists the specific assignment of final compact ID for each fabricated compact.

Table 1-1: Material identification record for A3-H08 compacts

Sample ID	Parent material	Notes
Blend F	Asbury 3482 natural graphite lot# 7602	Parent material information provided by
	Graftech GTI-D synthetic graphite CAS# 7782-42-5	INL
	Hexion D_SD-1708 Batch# 347715	
	Hexamethylenetetramine	
A3-H08-#	Blend F	Ten cylinders, A3-H08-1 through
		A3-H08-10, pressed from Blend F and
		carbonized
A3-H08-#-A	A3-H08-#	Two compacts machined from each of ten
and		carbonized cylinders, A3-H08-1 through
A3-H08-#-B		A3-H08-10
A3-H08-Z##	A3-H08-#-A	Twenty heat-treated compacts
	and	randomized and relabeled, A3-H08-Z01
	A3-H08-#-B	through A3-H08-Z20 (see DRF-39B for
		specific assignment record)

Table 1-2: Disposition of A3-H08 compacts

Sent to INL	Sent to Shiva		Retained at ORNL	
A3-H08-Z07	A3-H08-Z02	A3-H08-Z04	A3-H08-Z10	A3-H08-Z16
A3-H08-Z19	A3-H08-Z03	A3-H08-Z05	A3-H08-Z11	A3-H08-Z17
A3-H08-Z01 (spare)	A3-H08-Z15	A3-H08-Z06	A3-H08-Z12	A3-H08-Z18
		A3-H08-Z08	A3-H08-Z13	A3-H08-Z20
		A3-H08-Z09	A3-H08-Z14	

Table 1-3. Record of assignment of Z-number to fabricated compacts (DRF-39B)

Data Report Form D	RF-39B: Compact Tracking
Procedure:	AGR-CHAR-DAM-39 Rev. 0
Operator:	John Hunn
Compact lot ID:	A3-H08
Compact Lot description:	Matrix-only compacts from Hexion D_SD-1708+7.5% HEXA
Filename:	\\mc-agr\AGR\CompactDimensions\A3-H08_DRF39R0.xls

Compact Z Number	Compact Fab Number
Z01	7-B
Z02	6-B
Z03	4-A
Z04	10-A
Z05	9-B

Compact Z Number	Compact Fab Number
Z06	9-A
Z07	3-B
Z08	8-A
Z09	6-A
Z10	3-A

Operator

Compact Z Number	Compact Fab Number
Z11	2-A
Z12	10-B
Z13	1-A
Z14	1-B
Z15	7-A

Compact Z Number	Compact Fab Number	
Z16	5-A	
Z17	2-B	
Z18	4-B	
Z19	5-B	
Z20	8-B	

	Comments	
1 11	10-27-10	
John Am	71 0 - 1 -	

2 <u>Inspection of Length, Diameter, and Matrix Density</u>

At the end of this section is the data report form DRF-39A associated with the compact lot A3-H08. This data report form summarizes the acceptance testing performed according to the product inspection plan AGR-CHAR-PIP-16. The information in this form covers all the property specifications listed in sections 2.03 and 2.04 of SPC-1285. All compacts met the specified criteria for diameter and matrix density. Six compacts did not meet the specified criteria for length. These compacts were not used. All compacts selected for irradiation or impurity analysis met all dimensional and density specifications. Note that the specification only requires compacts used for irradiation meet these requirements, so the final determination of this inspection is that the product (compacts used for irradiation) conforms to the specified requirements for length, diameter, and density.

Table 2-1 summarizes the critical properties of the compacts selected for possible insertion in the AGC-2 irradiation experiment. Table 2-2 is a copy of data report form DRF-39A.

Table 2-1. Summary of properties of compact shipped for irradiation

Compact ID	Length (mm)	Diameter (mm)	Density (g/cm³)
A3-H08-Z07	6.314	12.73	1.694
A3-H08-Z19	6.305	12.72	1.673
A3-H08-Z01 (spare)	6.310	12.72	1.676
Specification	$6.299 \le x \le 6.350$	$12.700 \le x \le 12.751$	1.75 ± 0.13

Table 2-2. Record of compact acceptance test for dimensions and density (DRF-39A)

Procedure	AGR-CHAR-DAM-39 Rev. 0
Operator	John Hunn
Compact lot ID:	A3-H08
Compact Lot description:	Matrix-only compacts from Hexion D_SD-1708+7.5% HEX
Filename	\mc-agr\AGR\CompactDimensions\A3-H08_DRF39R0.xls
Vertical height gauge calibration due date	3/22/11
Digital caliper calibration due date	5/11/11
Gauge blocks calibration due date:	11/7/12
Analytical balance calibration due date:	9/13/11

Acceptance criteria for compact length: ≥6.299 and ≤6.350 mm	
Acceptance criteria for compact diameter: ≥12.700 and ≤12.751 mm	
Acceptance criteria for compact mass: For information only	
Acceptance criteria for compact density: 1.75±0.13 g/cm ³	

Compact	Mass	Length	-	Diameter (mm)		Volume	Density	Accept?	
ID Number	(g)	(mm)	0°	90°	Average	(cm ³)	(g/cm³)	(pass or fail)	
Z01	1.3441	6.310	12.72	12.72	12.72	0.8019	1.6762	pass	
Z02	1.3549	6.334	12.73	12.73	12,73	0.8062	1.6807	pass	
Z03	1.3682	6.299	12.71	12.71	12.71	0.7992	1.7120	pass	
Z04	1.3662	6.314	12.74	12.74	12.74	0.8049	1.6974	pass	
Z05	1.3619	6.358	12.73	12.73	12.73	0.8092	1.6830	fall	
Z06	1.4249	6.330	12.74	12.74	12.74	0.8069	1.7658	pass	
Z07	1.3612	6.314	12.73	12.73	12.73	0.8036	1.6938	pass	
Z08	1.4506	6.342	12.75	12.75	12.75	0.8097	1.7915	pass	
Z09	1.4463	6.353	12.74	12.74	12.74	0.8099	1.7859	fall	
Z10	1.4324	6,367	12.74	12.74	12.74	0.8116	1.7648	fail	
Z11	1.3427	6.305	12.73	12.73	12.73	0.8025	1.6732	pass	
Z12	1.3775	6.337	12.73	12.74	12.74	0.8072	1.7066	pass	
Z13	1.4236	6.357	12.74	12.74	12.74	0.8104	1,7567	fall	
Z14	1.3729	6.342	12.74	12.74	12.74	0.8085	1.6982	pass	
Z15	1.3914	6.337	12.73	12.73	12.73	0.8065	1.7251	pass	
Z16	1.4381	6.352	12.74	12.74	12.74	0.8097	1.7760	fall	
217	1.3640	6.353	12.74	12.74	12.74	0.8099	1.6843	fall	
Z18	1.3626	6.311	12,71	12.71	12.71	0.8007	1.7017	pass	
Z19	1.3390	6.305	12.71	12.72	12.72	0.8006	1,6725	pass	
Z20	1.3684	6.342	12.74	12.74	12.74	0.8085	1.6926	pass	

John Hm	10-27-10
Operator	Date
John Rhim	10-28-10
QC Supervisor	Date

3 Inspection of Compact Surface Appearance

All but 3 compacts had obvious surface fissures on the sides (Figure 3-1 and Figure 3-2) and surface damage on one or both ends (Figure 3-3). Two of the compacts that did not have obvious surface fissures were selected for irradiation (see Table 1-2). These compacts are shown in Figure 3-4 through Figure 3-9. Note that both compacts showed noticeable spiral machining artifacts on one end. The third that did not have obvious surface fissures was selected as a spare; however, this third compact had visible scarring from machining in a band around the side (Figure 3-10).

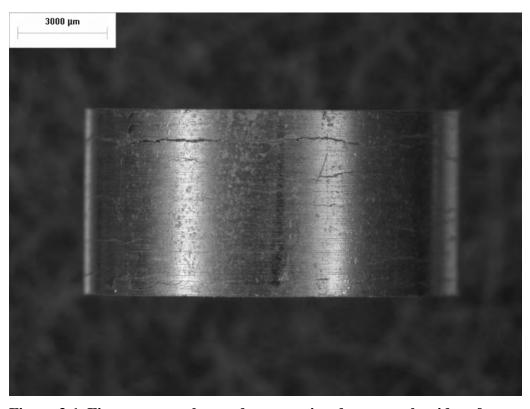


Figure 3-1. Fissures were observed to a varying degree on the sides of most compacts. Compact A3-H08-Z04 is shown.

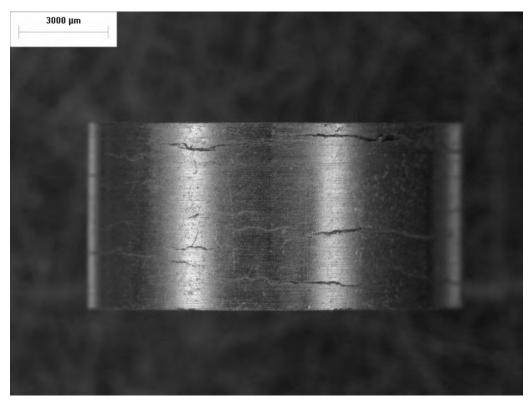


Figure 3-2. Another rotation of compact A3-H08-Z04 showing side fissures.

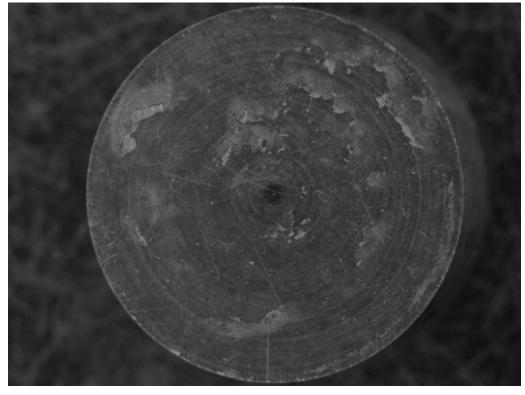


Figure 3-3. End faces of many compacts showed damage that looked like flakes of material were delaminating and sometimes breaking away. Compact A3-H08-Z04 is shown.

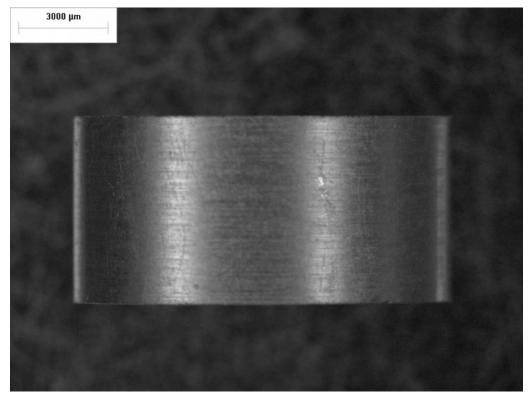


Figure 3-4. No obvious side fissures were observed on A3-H08-Z07.

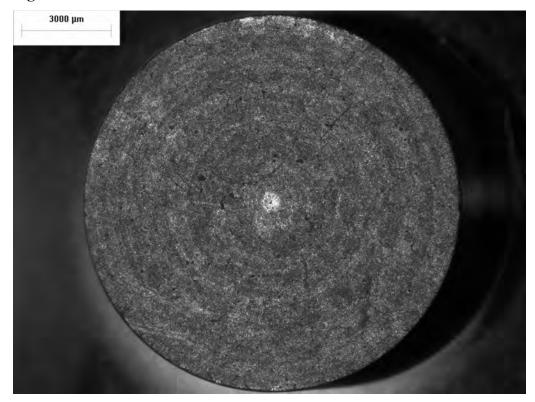


Figure 3-5. One end of A3-H08-Z07.

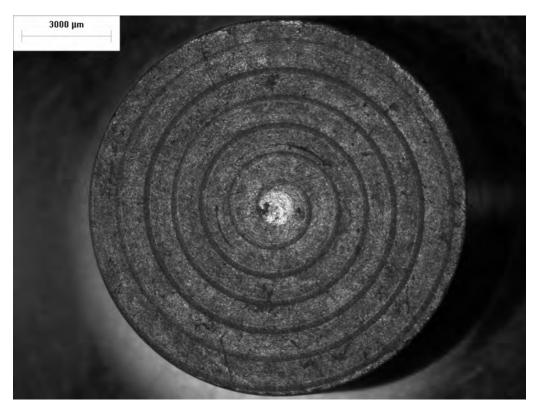


Figure 3-6. Other end of A3-H08-Z07. A spiral machining artifact was observed.

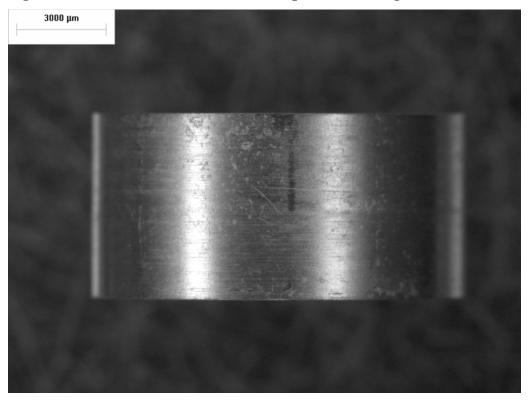


Figure 3-7. No obvious side fissures were observed on A3-H08-Z19.

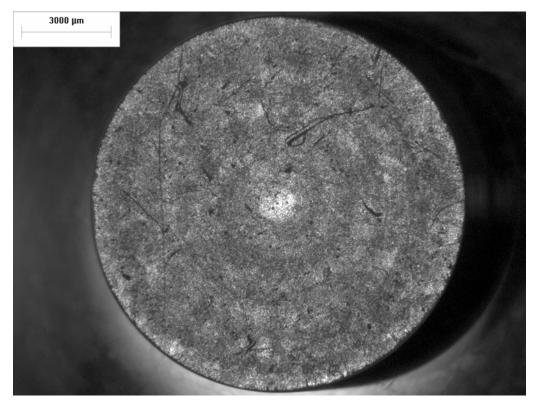


Figure 3-8. One end of A3-H08-Z19.

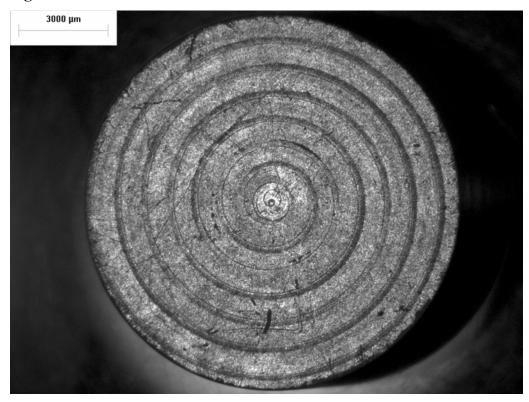


Figure 3-9. Other end of A3-H08-Z19. A spiral machining artifact was observed.

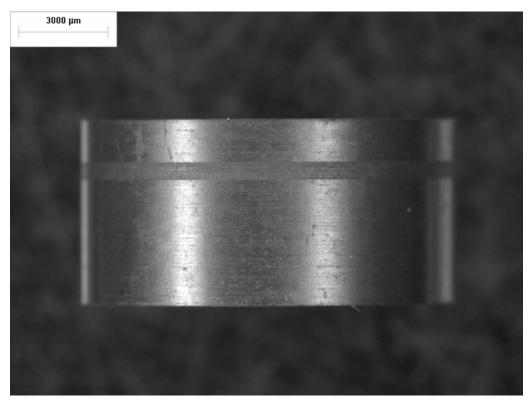


Figure 3-10. Machining artifact on A3-H08-Z01. No side fissures were visible.

4 Compacting process conditions

Initial matrix-only compacting tests were performed using parameters suggested by INL, which were selected based on manufacturer provided resin properties and previous experiments with overcoated material. These test compacts exhibited problems including delamination or fracture during ejection from the die and development of fissures during carbonization. The higher fraction of resin in the matrix-only compacts compared to compacts made from overcoated particles may have been a contributing factor to the difficulties encountered. To reduce the observed cracking, additional development was carried out before the final matrix-only compacts were fabricated. As part of this development, several compacts were made at various pressing conditions to determine the most effective fabrication methods. A summary of the final fabrication procedure is outlined below. This procedure was able to produce compacted cylinders with minimal visible flaws in the as-compacted (green) state. However, fissures still developed during carbonization.

AGC-2 matrix-only compacts needed to be nominally 12.7 mm in diameter, 6.35 mm in length, and 1.75 g/cm³ in density. Normally, a custom die would be made to produce compacts with the required dimensions. However, a limited time was available for fabrication of these compacts in order to have them available for inclusion in the AGC-2 irradiation test. The required fabrication schedule made it infeasible to obtain a new compacting die with a diameter that would yield the desired final compact diameter after heat-treatment. As an alternative approach, a slightly oversized (15.24 mm diameter) die was used to produce 10 cylinders from each matrix blend that were then machined into the final 20 compacts. The die was single-acting, that is, the top punch moved and the bottom punch was fixed. To obtain two compacts from each carbonized cylinder, a minimum length of at least 25.4 mm long was desired to allow for gripping of the cylinder and machining loss. However, the compacting die, which had ~75 mm of available length for loading, was not tall enough to hold the amount of as-received matrix precursor powder required to produce a compacted cylinder at least 25.4 mm long. The jet milled powder had a low bulk density, so a pre-compacting method for the as-received powder was developed to allow the required amount of feed material to fit in the available die. Another die (25.4 mm diameter) was used to produce ~1 g/cm³ slugs, which were re-granulated into a coarse powder that would work with the 15.24 mm diameter compacting die. These low density slugs were made by filling the 25.4 mm diameter die at room temperature with the as-received matrix precursor blend and pressing to 8 kN. The slugs were then broken into granules and passed through a U.S. Standard #20 (850 μ m nominal opening) sieve to remove any large chunks.

Table 4-1 provides a summary of the critical steps in the compacting schedule. All AGC-2 matrix-only compacts were made using standard operating procedure AGR-COMP-SOP-10, "Production of AGC Irradiation Specimens" and the following pressing schedule. Using the precompacting method described above, charges were prepared and weighed out while the compacting die, top punch, and bottom punch were heated simultaneously to 130±1°C. The compact charge weight was calculated to produce a compact of the required length and density, based on empirical data on weight loss and shrinkage. During heating, the inner diameter and contact surfaces of the top and bottom punches were lightly sprayed with McLube 860 mold release agent and allowed to dry. The charge was poured into the compacting die within an approximately 30 second long period. The top punch was put in place and the matrix precursor

material was allowed to warm up in the die for an additional 20-30 seconds before the programmed compacting sequence was initiated on the Promess press. The press ram was advanced at 1 mm/sec, taking approximately 20 seconds to reach the target position. This target position was chosen to produce a compact of the desired length. At 1 mm short of the target position, there was a 1 second delay. This delay provided additional relaxation time for the material and reduced the peak force applied during compacting. After the 1 second delay, the press continued to move to the target position. Once at the target position, the press held position for 60 seconds. After the hold, the press retracted and an ejection block was set in place to allow the lower punch and cylinder to be ejected from the bottom of the die.

Table 4-1. Compacting schedule summary

Step	Action	Timing	Comments
1	Load die	~30 sec to load	
2	Delay	~30 sec	Allow matrix precursor material to heat up
3	Initiate program		
4	Move to position	~20 sec	Pressing at 1 mm/sec
5	Delay	1 sec	Hold position 1 mm short of target position
6	Move to position	1 sec	Continue to target position at 1mm/sec
7	Delay	60 sec	Hold position
8	Move to position	N/A	Retract for ejection
9	Move to position	N/A	Eject part at 3 mm/sec

The compacted cylinders were carbonized per procedure AGR-COMP-SOP-04, "Carbonizing Compacts" by heating to 950°C at a rate of 1°C/min in flowing helium, followed by a 1 hour hold. The carbonized cylinders were machined according to drawing AGR-COMP-DWG-03, "AGC Compact Drawing" (see appendix A). Cylinders were first turned down to the target diameter. Prior to cutting two compacts of the target length from each cylinder, a thin layer of material was removed from the end to square the cylinder. The two machined compacts were identified by adding "A" or "B" to the fabrication ID relative to their location within the original compact (A being closest to the top of the die). Most of the B compacts came from near the middle of the cylinder. However, some of the compacted cylinders fractured during carbonization (see Table 4-2). For these cylinders, the B compact was taken from closer to the bottom of the cylinder. After machining, the compacts were heat-treated per procedure AGR-COMP-SOP-05, "Heat-treating Compacts" by heating to 1800°C under vacuum at a rate of 20°C/min, followed by a 1 hour hold. The main purpose of the heat-treatment process was to reduce impurity content in the compacts. Compacts were heat-treated after machining to reduce the potential for contamination after the heat-treatment step.

Based on previous experience with AGR matrix materials, most dimensional changes in the pressed cylinders were expected to occur during carbonization. However, compacts can also undergo dimensional changes during heat-treatment. These changes are usually a consistent minor shrinkage and can be compensated for by using empirical data to oversize the compacts. During initial testing, it was determined that the dimensional change induced by the heat-treatment of the A3-H08 matrix material was inconsistent. Some test compacts shrank, while others grew in length. For this reason, compacts were machined to a length close to the middle of the specified final length for the heat-treated compacts. This provided the most flexibility and

allowed for re-machining of compacts that were too long. Upon inspection of the heat-treated compacts, there were enough compacts with an acceptable length, so re-machining was not necessary.

Table 4-2 provides dimensional analysis for the cylinders before and after carbonization. Table 4-3 provides dimensional analysis for the machined compacts before and after heat-treatment. Table 4-2 also lists the compaction force required to press each green compact. Average density was determined from the measured weight and dimensions assuming a cylindrical geometry. Some cylinders could not be measured after carbonization because they fractured during the carbonization process.

Because of the presence of fissures in the cylinders and compacts, it is difficult to draw conclusions from the analysis of the data in Table 4-2 and Table 4-3. These fissures were visible in most of the final heat-treated compacts and are discussed in section 3. These fissures can add to the length of the compact and produce an error in the calculated density.

Fractional weight loss after carbonization was consistent in all the cylinders that were measured. Fractional change in length after carbonization was less consistent due to the presence of fissures in the cylinders. Table 4-3 shows that many of the machined compacts were longer after heat-treatment. This was also most likely due to the formation or expansion of these fissures. There was also a slight weight loss after heat-treatment, except for one compact which inexplicably gained weight. In general, the calculated density of the compacts taken from closer to the top of the cylinder was higher than those taken from lower down because the compacting force was greater closer to the moving punch.

Table 4-2. Analysis of cylinders before and after carbonization

		Green				Carbonized				Fractional cl	hange from g	reen to carb	onized
Fabrication ID	Compaction Force (Mpa)	Length (mm)	Dia (mm)	Mass (g)	Density (g/cm²)	Length (mm)	Dia (mm)	Mass (g)	Density (g/cm²)	Length	Dia	Mass	Density
A3-H08-1	23.5	26.30	15.27	8.4050	1.745	25.35	15.04	7.7402	1.719	0.964	0.985	0.921	0.985
A3-H08-2 A3-H08-3	26.2 24.6	25.97 26.20	15.30 15.30	8.4017 8.4080	1.760 1.745	25.17 N/A	15.01 N/A	7.7350 N/A	1.737 N/A	0.969 N/A	0.981 N/A	0.921 N/A	0.987 N/A
A3-H08-4 A3-H08-5 A3-H08-6	24.7 23.7	26.67 26.20	15.31 15.30	8.4071 8.4069	1.712 1.745 1.744	25.69 N/A	15.03 N/A	7.7357 N/A	1.697 N/A	0.963 N/A	0.982 N/A	0.920 N/A	0.991 N/A
A3-H08-7 A3-H08-8	23.9 26.3 23.7	26.20 26.36 26.06	15.31 15.30 15.31	8.4131 8.4100 8.4138	1.735 1.754	25.31 N/A 25.23	15.07 N/A 15.07	7.7460 N/A 7.7455	1.716 N/A 1.721	0.966 N/A 0.968	0.984 N/A 0.984	0.921 N/A 0.921	0.984 N/A 0.981
A3-H08-9 A3-H08-10	25.0 24.8	26.21 26.60	15.32 15.33	8.4123 8.4170	1.741 1.714	25.25 25.39 N/A	15.10 N/A	7.7394 N/A	1.702 N/A	0.969 N/A	0.986 N/A	0.921 0.920 N/A	0.978 N/A

N/A = Not applicable (carbonized length could not be determined because cylinder broke during carbonization)

Table 4-3: Analysis of machined compacts before and after heat-treatment

	Carbonized			Heat Treated				Fractional change from carbonized to heat treated					
Fabrication ID	Characterization ID	Length (mm)	Dia (mm)	Mass (g)	Density (g/cm²)	Length (mm)	Dia (mm)	Mass (g)	Density (g/cm²)	Length	Dia	Mass	Density
A2 1100 1 A	12 1100 712	6.24	12.72	1 1200	1 7740	6 257	12.74	1 4226	1 7567	1.003	1 002	0.006	0.000
A3-H08-1-A	A3-H08-Z13	6.34	12.72	1.4299	1.7748	6.357	12.74	1.4236	1.7567	1.003	1.002	0.996	0.990
A3-H08-1-B	A3-H08-Z14	6.34	12.73	1.3792	1.7092	6.342	12.74	1.3729	1.6982	1.000	1.001	0.995	0.994
A3-H08-2-A	A3-H08-Z11	6.31	12.72	1.3505	1.6842	6.305	12.73	1.3427	1.6732	0.999	1.001	0.994	0.993
A3-H08-2-B	A3-H08-Z17	6.32	12.72	1.3706	1.7066	6.353	12.74	1.3640	1.6843	1.005	1.002	0.995	0.987
A3-H08-3-A	A3-H08-Z10	6.32	12.72	1.4386	1.7913	6.367	12.74	1.4324	1.7648	1.007	1.002	0.996	0.985
A3-H08-3-B	A3-H08-Z07	6.31	12.72	1.3680	1.7061	6.314	12.73	1.3612	1.6938	1.001	1.001	0.995	0.993
A3-H08-4-A	A3-H08-Z03	6.32	12.72	1.3782	1.7161	6.299	12.71	1.3682	1.7120	0.997	0.999	0.993	0.998
A3-H08-4-B	A3-H08-Z18	6.33	12.72	1.3723	1.7060	6.311	12.71	1.3626	1.7017	0.997	0.999	0.993	0.997
A3-H08-5-A	A3-H08-Z16	6.33	12.72	1.4443	1.7955	6.352	12.74	1.4381	1.7760	1.003	1.002	0.996	0.989
A3-H08-5-B	A3-H08-Z19	6.31	12.72	1.3465	1.6792	6.305	12.71	1.3390	1.6738	0.999	0.999	0.994	0.997
A3-H08-6-A	A3-H08-Z09	6.33	12.73	1.4515	1.8016	6.353	12.74	1.4463	1.7859	1.004	1.001	0.996	0.991
A3-H08-6-B	A3-H08-Z02	6.32	12.72	1.3606	1.6941	6.334	12.73	1.3549	1.6807	1.002	1.001	0.996	0.992
A3-H08-7-A	A3-H08-Z15	6.32	12.72	1.3968	1.7392	6.337	12.73	1.3914	1.7251	1.003	1.001	0.996	0.992
A3-H08-7-B	A3-H08-Z01	6.32	12.72	1.3506	1.6817	6.310	12.72	1.3441	1.6762	0.998	1.000	0.995	0.997
						I							
A3-H08-8-A	A3-H08-Z08	6.31	12.73	1.4557	1.8126	6.342	12.75	1.4506	1.7915	1.005	1.002	0.996	0.988
A3-H08-8-B	A3-H08-Z20	6.32	12.72	1.3738	1.7106	6.342	12.74	1.3684	1.6926	1.003	1.002	0.996	0.989
A3-H08-9-A	A3-H08-Z06	6.31	12.73	1.4297	1.7802	6.330	12.74	1.4249	1.7658	1.003	1.001	0.997	0.992
A3-H08-9-B	A3-H08-Z05	6.34	12.72	1.3676	1.6975	6.358	12.73	1.3619	1.6830	1.003	1.001	0.996	0.991
A3-H08-10-A	A3-H08-Z04	6.31	12.73	1.3831	1.7222	6.314	12.74	1.3662	1.6974	1.001	1.001	0.988	0.986
A3-H08-10-B	A3-H08-Z12	6.33	12.72	1.3716	1.7051	6.337	12.73	1.3775	1.7079	1.001	1.001	1.004	1.002

5 Impurity analysis of matrix, resin, and graphite

Samples of the natural and synthetic graphite, resin, and matrix precursor blend F were sent to Shiva Technologies for impurity analysis. The matrix precursor powder for compact lot A3-H08 was a jet milled blend of 64 wt% natural graphite (Asbury 3482), 16 wt% synthetic graphite (Graftech GTI-D), and 20 wt% high purity novolac resin (Hexion D_SD-1708 with 7.5% hexamethylenetetramine added). The resin and matrix precursor blend were carbonized prior to shipment to Shiva. The carbonization process was used to remove volatiles that would interfere with the impurity analysis. Approximately 5 grams of resin and graphite/resin blend were loaded into separate quartz trays and then placed into a tube furnace. The samples were heated to 950°C at a rate of 5°C/min and held at 950°C for 1 hour. The furnace power was cut and the samples cooled to room temperature. The samples were then loaded into glass vials and shipped to SHIVA for full scan glow discharge mass spectrometry (GDMS) analysis, along with samples of the natural and synthetic graphite.

Table 5-1 is a compilation of the impurity analysis results. Impurities that could not be detected above the analysis threshold value are reported as less than values (<) and appear in gray. Values marked as less than or equal to (=<) indicate that a measurable value of the element was obtained, but that this element may have come from the Shiva sample preparation. Prior to GDMS analysis, Shiva poured the powder samples into a Teflon mold with a binder material. The binder was mostly indium, but may also have contained other elements, as indicated in the analysis report by the "=<" symbol. The resin showed very high values for F, but these were marked as semiquantitative (~) and probably came from the Teflon mold. The natural graphite was very high in Si and also high in Mg, Al, S, and Fe. The synthetic graphite was high in Cu. The impurities in the graphite carried over to the jet milled blend. The hexamethylenetetramine (hexa) was high in several impurities, but these did not significantly impact the matrix precursor blend.

Copies of the Shiva Technologies certified impurity analysis data sheets for the natural and synthetic graphite are provided in Table 5-2 and Table 5-3. Copies of the certified impurity analysis data sheets for the Hexion resin and hexamethylenetetramine are provided in Table 5-4 and Table 5-5. A copy of the certified impurity analysis data sheet for the graphite/resin blend made from these materials (INL Blend F) is provided in Table 5-6.

Samples of the final heat treated compacts were also sent to Shiva for analysis. These results are presented in section 6. As discussed in that section, heat-treatment of the compacts removed some of the impurities present in the graphite/resin blend.

Table 5-1. Summary of feedstock impurities

El 4	Concentration (ppm wt)								
Element	Asbury 3482 GTI-D Hexion SD-1708 Hexa								
Li	< 0.01	< 0.01	0.5	6.4	0.2				
Be	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01				
В	0.24	0.77	0.06	2.5	0.4				
С	Matrix	Matrix	Matrix	Matrix	Matrix				
N	-	-	-	-	-				
О	-	-	-	-	-				
F	~ 20	~ 10	~ 560	~ 960	~ 10				
Na	1.5	11	5.4	220	2				
Mg	80	1.2	< 0.5	28	68				
Al	29	0.9	8	16	30				
Si	710	2.7	18	130	700				
P	0.82	< 0.1	0.25	4.8	0.9				
S	25	10	3.5	44	12				
Cl	2.5	7.8	0.79	3.8	7.5				
K	0.2	< 0.1	0.2	5.4	< 0.1				
Ca	8.2	3.2	4.2	33	15				
Sc	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05				
Ti	2.3	2.2	0.3	5.3	1.7				
V	0.45	9.9	0.02	0.04	1.7				
Cr	< 0.5	< 0.5	3.9	< 0.5	< 0.5				
Mn	0.55	< 0.05	0.34	0.55	0.9				
Fe	38	1.1	18	39	50				
Со	< 0.05	< 0.05	1.6	< 0.05	0.12				
Ni	0.25	0.25	2.9	1.7	2.5				
Cu	2.5	120	21	42	32				
Zn	0.4	< 0.1	=< 2	220	0.3				
Ga	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1				
Ge	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1				
As	< 0.1	< 0.1	< 0.1	0.29	< 0.1				
Se	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1				
Br	0.41	< 0.1	< 0.1	< 0.1	1.4				
Rb	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05				
Sr	0.08	< 0.05	< 0.05	0.16	< 0.05				
Y	0.2	< 0.05	0.68	< 0.05	0.2				
Zr	0.6	0.14	< 0.05	< 0.05	2.2				
Nb	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1				
Mo	< 0.05	0.09	0.64	1.5	0.32				
Ru	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1				
Rh	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1				
Pd	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1				
Ag	< 0.1	< 0.1	< 0.1	3.9	< 0.1				
Cd	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1				
In	Binder	Binder	Binder	Binder	Binder				

Table 5-1. Summary of feedstock impurities (continued)

TI .	Concentration (ppm wt)									
Element	Asbury 3482	GTI-D	Hexion SD-1708	Hexa	INL Blend F					
Sn	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5					
Sb	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5					
Te	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1					
I	< 20	=< 80	=< 60	=< 550	< 20					
Cs	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1					
Ba	0.8	< 0.1	< 0.1	< 0.1	1.5					
La	< 0.5	=< 2	=< 6	=< 26	< 0.5					
Ce	< 0.5	< 0.5	< 0.5	=< 1.2	< 0.5					
Pr	< 0.05	< 0.05	=< 1.5	=< 2.8	< 0.05					
Nd	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05					
Sm	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05					
Eu	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05					
Gd	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05					
Tb	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05					
Dy	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05					
Но	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05					
Er	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05					
Tm	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05					
Yb	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05					
Lu	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05					
Hf	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05					
Ta	< 5	< 5	< 5	< 5	< 5					
W	0.15	< 0.05	0.08	1.2	0.17					
Re	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05					
Os	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05					
Ir	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05					
Pt	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05					
Au	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1					
Hg	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5					
T1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1					
Pb	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5					
Bi	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1					
Th	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05					
U	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05					

Table 5-2. Impurity analysis report for Asbury 3482 natural graphite lot# 7602



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SHIVA Technologies An Operating Unit of Evans Analytical Group LLC 6707 Brooklawn Parkway Syracuse, New York 13211 Telephone (315) 431-9900 Fax: (315) 431-9800 Email info.ny@eaglabs.com www.eaglabs.com

Customer: UT-Battelle Oak Ridge

P.O.# 4000100480

1 Bethel Valley Rd, Oak Ridge, TN 37823-6063 USA

.....

Date: 6-Nov-10 Customer ID: **C**

Job # S0ABV776 Sample ID: S101101057

Asbury 3482 NFG

Element	Concentration [ppm wt]	Element	Concentration [ppm wt]
Li	< 0.01	Pd	< 0.1
Ве	< 0.01	Ag	< 0.1
В	0.24	Cd	< 0.1
С	Matrix	ln	Binder
N	-	Sn	< 0.5
0	_	Sb	< 0.5
F	~ 20	Те	< 0.1
Na	1.5		< 20
Mg	80	Cs	< 0.1
Al	29	Ва	0.8
Si	710	La	< 0.5
P	0.82	Ce	< 0.5
S	25	Pr	< 0.05
CI	2.5	Nd	< 0.05
K	0.2	Sm	< 0.05
Ca	8.2	Eu	< 0.05
Sc	< 0.05	Gd	< 0.05
Ti	2.3	Tb	< 0.05
V	0.45	Dy	< 0.05
Cr	< 0.5	Ho	< 0.05
Mn	0.55	Er	< 0.05
Fe	38	Tm	< 0.05
Co	< 0.05	Yb	< 0.05
Ni	0.25	Lu	< 0.05
Cu	2.5	Hf	< 0.05
Zn	0.4	Та	< 5
Ga	< 0.1	W	0.15
Ge	< 0.1	Re	< 0.05
As	< 0.1	Os	< 0.05
Se	< 0.1	lr	< 0.05
Br	0.41	Pt	< 0.05
Rb	< 0.05	Au	< 0.1
Sr	0.08	Hg	< 0.5
Υ	0.2	ΤΪ	< 0.1
Zr	0.6	Pb	< 0.5
Nb	< 0.1	Bi	< 0.1
Mo	< 0.05	Th	< 0.05
Ru	< 0.1	U	< 0.05
Rh	< 0.1		

[~] Semiquantitative Values

J.SCHIEBLER (Analyst)

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Table 5-3. Impurity analysis report for Graftech GTI_D synthetic graphite #7782-42-5



Date:

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Customer: UT-Battelle Oak Ridge

P.O.# 4000100480

1 Bethel Valley Rd, Oak Ridge, TN 37823-6063 USA 6-Nov-10

Job # S0ABV776

Customer ID: C

Sample ID: \$101101058

GTI-D-SG

Element	Concentration [ppm wt]	Element	Concentration [ppm wt]
Li	< 0.01	Pd	< 0.1
Be	< 0.01	Ag	< 0.1
В	0.77	Cd	< 0.1
С	Matrix	In	Binder
N	-	Sn	< 0.5
0	-	Sb	< 0.5
F	~ 10	Te	< 0.1
Na	11	ı	=< 80
Mg	1.2	Cs	< 0.1
Al	0.9	Ва	< 0.1
Si	2.7	La	=< 2
Р	< 0.1	Ce	< 0.5
S	10	Pr	< 0.05
CI	7.8	Nd	< 0.05
K	< 0.1	Sm	< 0.05
Ca	3.2	Eu	< 0.05
Sc	< 0.05	Gd	< 0.05
Ti	2.2	Tb	< 0.05
V	9.9	Dy	< 0.05
Cr	< 0.5	Но	< 0.05
Mn	< 0.05	Er	< 0.05
Fe	1.1	Tm	< 0.05
Co	< 0.05	Yb	< 0.05
Ni	0.25	Lu	< 0.05
Cu	120	Hf	< 0.05
Zn	< 0.1	Та	< 5
Ga	< 0.1	W	< 0.05
Ge	< 0.1	Re	< 0.05
As	< 0.1	Os	< 0.05
Se	< 0.1	lr	< 0.05
Br	< 0.1	Pt	< 0.05
Rb	< 0.05	Au	< 0.1
Sr	< 0.05	Hg	< 0.5
Y	< 0.05	TI	< 0.1
Zr	0.14	Pb	< 0.5
Nb	< 0.1	Bi	< 0.1
Mo	0.09	Th	< 0.05
Ru	< 0.1	U	< 0.05
Rh	< 0.1		

[~] Semiquantitative Values

J.SCHIEBLER (Analyst)

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Table 5-4. Impurity analysis report for Hexion SD-1708 batch# 347715

1 Bethel Valley Rd, Oak Ridge, TN 37823-6063 USA



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Telephone (315) 431-9900 Fax: (315) 431-9800 Email info.ny@eaglabs.com www.eaglabs.com

Customer: **UT-Battelle Oak Ridge** P.O.#

4000100480

Date: 6-Nov-10 Job#

S0ABV776

Customer ID: C

Sample ID:

S101101061

Hexion SD-1708

Element	Concentration [ppm wt]	Element	Concentration [ppm wt]
Li	0.5	Pd	< 0.1
Be	< 0.01	Ag	< 0.1
В	0.06	Cd	< 0.1
С	Matrix	In	Binder
N	-	Sn	< 0.5
0	-	Sb	< 0.5
F	~ 560	Те	< 0.1
Na	5.4		=< 60
Mg	< 0.5	Cs	< 0.1
Al	8	Ва	< 0.1
Si	18	La	=< 6
Р	0.25	Ce	< 0.5
S	3.5	Pr	=< 1.5
Cl	0.79	Nd	< 0.05
K	0.2 4.2	Sm	< 0.05
Ca	4.2	Eu	< 0.05
Sc	< 0.05	Gd	< 0.05
Ti	0.3	Tb	< 0.05
V	0.02	Dy	< 0.05
Cr	3.9	Но	< 0.05
Mn	0.34	Er	< 0.05
Fe	18	Tm	< 0.05
Co	1.6	Yb	< 0.05
Ni	2.9	Lu	< 0.05
Cu	21	Hf	< 0.05
Zn	=< 2	Та	< 5
Ga	< 0.1	W	0.08
Ge	< 0.1	Re	< 0.05
As	< 0.1	Os	< 0.05
Se	< 0.1	lr	< 0.05
Br	< 0.1	Pt	< 0.05
Rb	< 0.05	Au	< 0.1
Sr	< 0.05	Hg	< 0.5
Y	0.68	TI	< 0.1
Zr	< 0.05	Pb	< 0.5
Nb	< 0.1	Bi	< 0.1
Mo	0.64	Th	< 0.05
Ru	< 0.1	U	< 0.05
Rh	< 0.1		

[~] Semiquantitative Values

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Table 5-5. Impurity analysis report for hexamethylenetetramine



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Customer: UT-Battelle Oak Ridge

P.O.#

4000100480

1 Bethel Valley Rd, Oak Ridge, TN 37823-6063 USA Date: 6-Nov-10

Job #

S0ABV776

Customer ID: C

Sample ID:

S101101062

Hexa

Element	Concentration [ppm wt]	Element	Concentration [ppm wt]
Li	6.4	Pd	< 0.1
Ве	< 0.01	Ag	3.9
В	2.5	Cd	< 0.1
С	Matrix	ln	Binder
N	-	Sn	< 0.5
0	-	Sb	< 0.5
F	~ 960	Te	< 0.1
Na	220		=< 550
Mg	28	Cs	< 0.1
Al	16	Ва	< 0.1
Si	130	La	=< 26
Р	4.8	Ce	=< 1.2
S	44	Pr	=< 2.8
Cl	3.8	Nd	< 0.05
K	5.4	Sm	< 0.05
Ca	33	Eu	< 0.05
Sc	< 0.05	Gd	< 0.05
Ti	5.3	Tb	< 0.05
V	0.04	Dy	< 0.05
Cr	< 0.5	Но	< 0.05
Mn	0.55	Er	< 0.05
Fe	39	Tm	< 0.05
Co	< 0.05	Yb	< 0.05
Ni	1.7	Lu	< 0.05
Cu	42	Hf	< 0.05
Zn	220	Та	< 5
Ga	< 0.1	W	1.2
Ge	< 0.1	Re	< 0.05
As	0.29	Os	< 0.05
Se	< 0.1	lr	< 0.05
Br	< 0.1	Pt	< 0.05
Rb	< 0.05	Au	< 0.1
Sr	0.16	Hg	< 0.5
Υ	< 0.05	TI	< 0.1
Zr	< 0.05	Pb	< 0.5
Nb	< 0.1	Bi	< 0.1
Mo	1.5	Th	< 0.05
Ru	< 0.1	U	< 0.05
Rh	< 0.1		
~ Semiguantitativ	o Values		

[~] Semiquantitative Values

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Table 5-6. Impurity analysis report for INL graphite/resin Blend F



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Customer: UT-Battelle Oak Ridge

P.O.#

4000100480

1 Bethel Valley Rd, Oak Ridge, TN 37823-6063 USA

Date: 7-Nov-10

Job #

S0ABV776

Customer ID: C

Sample ID:

S101101065

INL Blend F

Li 0.2 Be < 0.01 B 0.4 C Matrix N - O - F ~ 10 Na 2 Mg 68	Pd Ag Cd In Sn	< 0.1 < 0.1 < 0.1 Binder
B 0.4 C Matrix N - O - F ~ 10 Na 2	Cd In Sn	< 0.1 Binder
C Matrix N - O - F ~ 10 Na 2	Cd In Sn	Binder
N - O - F ~ 10 Na 2	Sn	Binder
O - F ~ 10 Na 2		- 0 E
F ~ 10 Na 2		< 0.5
Na 2	Sb	< 0.5
	Te	< 0.1
Ma	1	< 20
Mg 68	Cs	< 0.1
Al 30	Ва	1.5
Si 700	La	< 0.5
P 0.9	Ce	< 0.5
S 12	Pr	< 0.05
CI 7.5	Nd	< 0.05
K < 0.1	Sm	< 0.05
Ca 15	Eu	< 0.05
Sc < 0.05	Gd	< 0.05
Ti 1.7	Tb	< 0.05
V 1.7	Dy	< 0.05
Cr < 0.5	Ho	< 0.05
Mn 0.9	Er	< 0.05
Fe 50	Tm	< 0.05
Co 0.12	Yb	< 0.05
Ni 2.5	Lu	< 0.05
Cu 32	Hf	< 0.05
Zn 0.3	Та	< 5
Ga < 0.1	W	0.17
Ge < 0.1	Re	< 0.05
As < 0.1	Os	< 0.05
Se < 0.1	lr	< 0.05
Br 1.4	Pt	< 0.05
Rb < 0.05	Au	< 0.1
Sr < 0.05	Hg	< 0.5
Y 0.2 Zr 2.2	ΤĬ	< 0.1
Zr 2.2	Pb	< 0.5
Nb < 0.1	Bi	< 0.1
Mo 0.32	Th	< 0.05
Ru < 0.1	U	< 0.05
Rh < 0.1		

[~] Semiquantitative Values

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6 Inspection of heat-treated compact impurities

Three heat-treated compacts were sent to Shiva Technologies for impurity analysis (see Table 1-2). Although not required by the specification, these compacts also met the requirements for diameter, length, and density placed on the compacts used for irradiation. Where possible within this limitation, compacts were selected that were machined from the same pressed cylinders as the compacts selected for irradiation.

Table 6-1 is a compilation of the impurity analysis results. The impurities in the feedstock materials were discussed in section 5. For comparison, Table 6-1 includes the impurity analysis for the matrix blend F used to make the A3-H08 compacts. Impurities that could not be detected above the analysis threshold value are reported as less than values (<) and appear in gray. There is no indication that the compacting process significantly increased any of the impurity levels in the heat-treated matrix-only compacts above what was already in the matrix precursor. Some impurities were significantly reduced by the heat-treatment (e.g., Mg, Cl, Fe, and Cu). Other impurities present at moderate levels in the graphite/resin blend were not affected (e.g., Al, S, and Ca). The very high Si content from the natural graphite was reduced by about 30% in the heat-treated compacts, but was still very high.

Copies of the Shiva Technologies certified impurity analysis data sheets for the three heat-treated compacts are provided in Table 6-2, Table 6-3, and Table 6-4.

Table 6-1. Summary of heat-treated compact impurities

T31 4	Concentration (ppm wt)						
Element	A3-H08-Z02 A3-H08-Z03 A3-H08-Z15 INL Blei						
Li	< 0.01	< 0.01	< 0.01	0.2			
Be	< 0.01	< 0.01	< 0.01	< 0.01			
В	0.19	0.41	0.1	0.4			
С	Matrix	Matrix	Matrix	Matrix			
N	-	-	-	-			
О	-	-	-	-			
F	< 1	< 1	< 1	~ 10			
Na	0.22	0.07	< 0.05	2			
Mg	2.3	6.7	9.4	68			
Al	21	26	22	30			
Si	460	480	480	700			
P	0.92	0.88	0.91	0.9			
S	12	12	10	12			
Cl	0.17	0.21	0.11	7.5			
K	0.24	< 0.1	< 0.1	< 0.1			
Ca	15	19	14	15			
Sc	< 0.05	< 0.05	< 0.05	< 0.05			
Ti	1.5	1.4	0.95	1.7			
V	0.95	1	0.62	1.7			
Cr	< 0.5	< 0.5	< 0.5	< 0.5			
Mn	< 0.05	< 0.05	< 0.05	0.9			
Fe	0.28	0.32	0.13	50			
Co	< 0.05	< 0.05	< 0.05	0.12			
Ni	< 0.1	< 0.1	< 0.1	2.5			
Cu	< 0.1	< 0.1	< 0.1	32			
Zn	< 0.1	< 0.1	< 0.1	0.3			
Ga	< 0.1	< 0.1	< 0.1	< 0.1			
Ge	< 0.1	< 0.1	< 0.1	< 0.1			
As	< 0.1	< 0.1	< 0.1	< 0.1			
Se	< 0.1	< 0.1	< 0.1	< 0.1			
Br	< 0.1	< 0.1	< 0.1	1.4			
Rb	< 0.05	< 0.05	< 0.05	< 0.05			
Sr	< 0.05	< 0.05	< 0.05	< 0.05			
Y	0.08	< 0.05	0.09	0.2			
Zr	1.5	1.3	1.2	2.2			
Nb	< 0.1	< 0.1	< 0.1	< 0.1			
Mo	0.22	0.28	0.14	0.32			
Ru	< 0.1	< 0.1	< 0.1	< 0.1			
Rh	< 0.1	< 0.1	< 0.1	< 0.1			
Pd	< 0.1	< 0.1	< 0.1	< 0.1			
Ag	< 0.1	< 0.1	< 0.1	< 0.1			
Cd	< 0.1	< 0.1	< 0.1	< 0.1			
In	< 0.1	< 0.1	< 0.1	Binder			

Table 6-1. Summary of heat-treated compact impurities (continued)

	Concentration (ppm wt)							
Element	A3-H08-Z02	A3-H08-Z03	A3-H08-Z15	INL Blend F				
Sn	< 0.5	< 0.5	< 0.5	< 0.5				
Sb	< 0.5	< 0.5	< 0.5	< 0.5				
Te	< 0.1	< 0.1	< 0.1	< 0.1				
Ι	< 0.1	< 0.1	< 0.1	< 20				
Cs	< 0.1	< 0.1	< 0.1	< 0.1				
Ba	0.24	< 0.1	0.2	1.5				
La	< 0.5	< 0.5	< 0.5	< 0.5				
Ce	< 0.05	< 0.05	< 0.05	< 0.5				
Pr	< 0.05	< 0.05	< 0.05	< 0.05				
Nd	< 0.05	< 0.05	< 0.05	< 0.05				
Sm	< 0.05	< 0.05	< 0.05	< 0.05				
Eu	< 0.05	< 0.05	< 0.05	< 0.05				
Gd	< 0.05	< 0.05	< 0.05	< 0.05				
Tb	< 0.05	< 0.05	< 0.05	< 0.05				
Dy	< 0.05	< 0.05	< 0.05	< 0.05				
Но	< 0.05	< 0.05	< 0.05	< 0.05				
Er	< 0.05	< 0.05	< 0.05	< 0.05				
Tm	< 0.05	< 0.05	< 0.05	< 0.05				
Yb	< 0.05	< 0.05	< 0.05	< 0.05				
Lu	< 0.05	< 0.05	< 0.05	< 0.05				
Hf	< 0.05	< 0.05	< 0.05	< 0.05				
Ta	< 5	< 5	< 5	< 5				
W	0.22	0.15	0.1	0.17				
Re	< 0.05	< 0.05	< 0.05	< 0.05				
Os	< 0.05	< 0.05	< 0.05	< 0.05				
Ir	< 0.05	< 0.05	< 0.05	< 0.05				
Pt	< 0.05	< 0.05	< 0.05	< 0.05				
Au	< 0.1	< 0.1	< 0.1	< 0.1				
Hg	< 0.5	< 0.5	< 0.5	< 0.5				
Tl	< 0.1	< 0.1	< 0.1	< 0.1				
Pb	< 0.5	< 0.5	< 0.5	< 0.5				
Bi	< 0.1	< 0.1	< 0.1	< 0.1				
Th	< 0.05	< 0.05	< 0.05	< 0.05				
U	< 0.05	< 0.05	< 0.05	< 0.05				

Table 6-2. Impurity analysis report for heat-treated compact A3-H08-Z02

1 Bethel Valley Rd, Oak Ridge, TN 37823-6063 USA



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ANALYTICAL REPORT

SHIVA Technologies An Operating Unit of Evans Analytical Group LLC 6707 Brooklawn Parkway Syracuse, New York 13211 Telephone (315) 431-9900 Fax: (315) 431-9800 Email info.ny@eaglabs.com www.eaglabs.com

Customer: UT-Battelle Oak Ridge

P.O.# 40

4000100480

Date: 7-Nov-10

Job #

S0ABV776

Customer ID: C

Sample ID:

S101101066

A3-H08-Z02

Element	Concentration [ppm wt]	Element Concentration [ppm wt]		
Li	< 0.01	Pd	< 0.1	
Be	< 0.01	Ag	< 0.1	
В	0.19	Cd	< 0.1	
С	Matrix	ln	< 0.1	
N	-	Sn	< 0.5	
0	-	Sb	< 0.5	
F	< 1	Te	< 0.1	
Na	0.22		< 0.1	
Mg	2.3	Cs	< 0.1	
Al	21	Ва	0.24	
Si	460	La	< 0.5	
Р	0.92	Ce	< 0.05	
S	12	Pr	< 0.05	
Cl	0.17	Nd	< 0.05	
K	0.24	Sm	< 0.05	
Ca	15	Eu	< 0.05	
Sc	< 0.05	Gd	< 0.05	
Ti	1.5	Tb	< 0.05	
V	0.95	Dy	< 0.05	
Cr	< 0.5	Ho	< 0.05	
Mn	< 0.05	Er	< 0.05	
Fe	0.28	Tm	< 0.05	
Со	< 0.05	Yb	< 0.05	
Ni	< 0.1	Lu	< 0.05	
Cu	< 0.1	Hf	< 0.05	
Zn	< 0.1	Та	< 5	
Ga	< 0.1	W	0.22	
Ge	< 0.1	Re	< 0.05	
As	< 0.1	Os	< 0.05	
Se	< 0.1	lr	< 0.05	
Br	< 0.1	Pt	< 0.05	
Rb	< 0.05	Au	< 0.1	
Sr	< 0.05	Hg	< 0.5	
Υ	0.08	ΤΪ	< 0.1	
Zr	1.5	Pb	< 0.5	
Nb	< 0.1	Bi	< 0.1	
Мо	0.22	Th	< 0.05	
Ru	< 0.1	U	< 0.05	
Rh	< 0.1			

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Table 6-3. Impurity analysis report for heat-treated compact A3-H08-Z03

1 Bethel Valley Rd, Oak Ridge, TN 37823-6063 USA



GDMS ANALYTICAL REPORT SHIVA Technologies An Operating Unit of Evans Analytical Group LLC 6707 Brooklawn Parkway Syracuse, New York 13211

Telephone (315) 431-9900 Fax: (315) 431-9800 Email info.ny@eaglabs.com www.eaglabs.com

Customer: **UT-Battelle Oak Ridge** P.O.#

4000100480

Date: 7-Nov-10

Job#

S0ABV776

Customer ID: C

Sample ID:

S101101067

A3-H08-Z03

Element	Concentration [ppm wt]	Element	Concentration [ppm wt]
Li	< 0.01	Pd	< 0.1
Be	< 0.01	Ag	< 0.1
В	0.41	Cd	< 0.1
С	Matrix	In	< 0.1
N	-	Sn	< 0.5
0	-	Sb	< 0.5
F	< 1	Те	< 0.1
Na	0.07		< 0.1
Mg	6.7	Cs	< 0.1
ΑĬ	26	Ва	< 0.1
Si	480	La	< 0.5
P	0.88	Ce	< 0.05
S	12	Pr	< 0.05
Cl	0.21	Nd	< 0.05
K	< 0.1	Sm	< 0.05
Ca	19	Eu	< 0.05
Sc	< 0.05	Gd	< 0.05
Ti	1.4	Tb	< 0.05
V	1	Dy	< 0.05
Cr	< 0.5	Ho	< 0.05
Mn	< 0.05	Er	< 0.05
Fe	0.32	Tm	< 0.05
Co	< 0.05	Yb	< 0.05
Ni	< 0.1	Lu	< 0.05
Cu	< 0.1	Hf	< 0.05
Zn	< 0.1	Та	< 5
Ga	< 0.1	W	0.15
Ge	< 0.1	Re	< 0.05
As	< 0.1	Os	< 0.05
Se	< 0.1	lr	< 0.05
Br	< 0.1	Pt	< 0.05
Rb	< 0.05	Au	< 0.1
Sr	< 0.05	Hg	< 0.5
Y	< 0.05	ΤΪ	< 0.1
Zr	1.3	Pb	< 0.5
Nb	< 0.1	Bi	< 0.1
Мо	0.28	Th	< 0.05
Ru	< 0.1	U	< 0.05
Rh	< 0.1	_	

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Table 6-4. Impurity analysis report for heat-treated compact A3-H08-Z15



Date:

GDMS
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SHIVA Technologies An Operating Unit of Evans Analytical Group LLC 6707 Brooklawn Parkway Syracuse, New York 13211 Telephone (315) 431-9900 Fax: (315) 431-9800 Email info.ny@eaglabs.com www.eaglabs.com

Customer: UT-Battelle Oak Ridge

P.O.# 4000100480

1 Bethel Valley Rd, Oak Ridge, TN 37823-6063 USA 7-Nov-10

Job #

S0ABV776

Customer ID: C

Sample ID:

S101101068

A3-H08-Z15

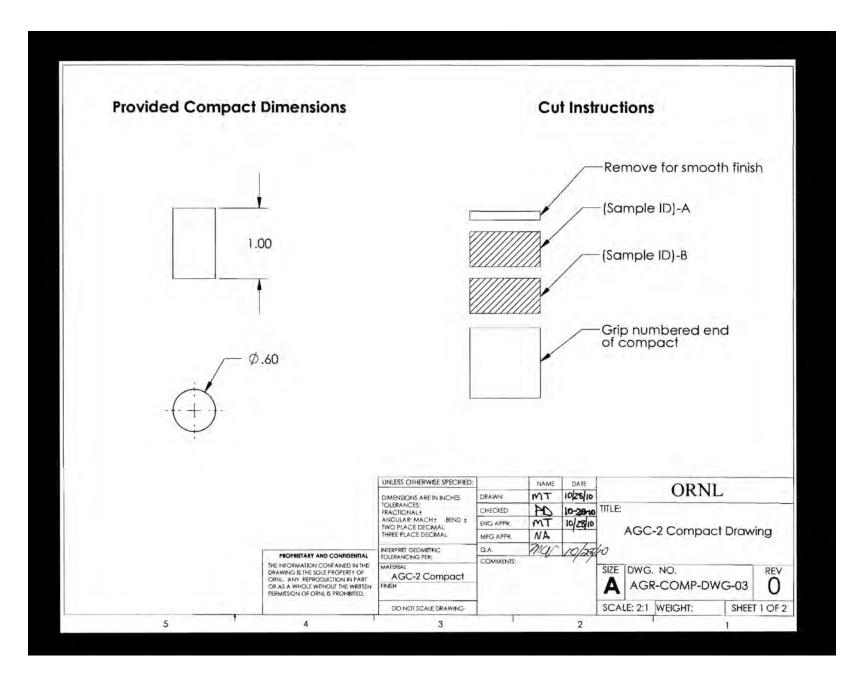
Li	Element	Concentration [ppm wt]	Element	Concentration [ppm wt]	
B 0.1 Cd < 0.1 C Matrix In < 0.1	Li	< 0.01	Pd	< 0.1	
B 0.1 Cd < 0.1 C Matrix In < 0.1	Be	< 0.01	Ag	< 0.1	
N - Sn < 0.5 O - Sb < 0.5	В		Cd	< 0.1	
N - Sn < 0.5 O - Sb < 0.5	С	Matrix	ln	< 0.1	
O - Sb < 0.5 F < 1		-			
F < 1 Te < 0.1 Na < 0.05		-	Sb		
Na < 0.05 I < 0.1 Mg 9.4 Cs < 0.1	F	< 1		< 0.1	
AI 22 Ba 0.2 Si 480 La < 0.5	Na	< 0.05		< 0.1	
AI 22 Ba 0.2 Si 480 La < 0.5	Mg	9.4	Cs	< 0.1	
P 0.91 Ce < 0.05 S 10 Pr < 0.05		22		0.2	
P 0.91 Ce < 0.05 S 10 Pr < 0.05	Si	480	La	< 0.5	
S 10 Pr < 0.05 CI 0.11 Nd < 0.05		0.91	Ce	< 0.05	
CI 0.11 Nd < 0.05 K < 0.1	S		Pr		
K < 0.1 Sm < 0.05 Ca 14 Eu < 0.05		0.11	Nd		
Sc < 0.05 Gd < 0.05 Ti 0.95 Tb < 0.05	K	< 0.1			
Ti 0.95 Tb < 0.05 V 0.62 Dy < 0.05	Ca	14	Eu	< 0.05	
Ti 0.95 Tb < 0.05 V 0.62 Dy < 0.05	Sc	< 0.05	Gd	< 0.05	
V 0.62 Dy < 0.05 Cr < 0.5		0.95	Tb	< 0.05	
Mn < 0.05 Er < 0.05 Fe 0.13 Tm < 0.05	V		Dy		
Mn < 0.05 Er < 0.05 Fe 0.13 Tm < 0.05	Cr	< 0.5	Ho	< 0.05	
Fe 0.13 Tm < 0.05 Co < 0.05	Mn		Er	< 0.05	
Co < 0.05 Yb < 0.05 Ni < 0.1	Fe		Tm		
Cu < 0.1 Hf < 0.05 Zn < 0.1		< 0.05	Yb	< 0.05	
Zn < 0.1 Ta < 5 Ga < 0.1	Ni	< 0.1	Lu	< 0.05	
Ga < 0.1 W 0.1 Ge < 0.1	Cu	< 0.1	Hf	< 0.05	
Ge < 0.1 Re < 0.05 As < 0.1	Zn	< 0.1	Та	< 5	
As < 0.1 Os < 0.05 Se < 0.1	Ga	< 0.1	W	0.1	
Se < 0.1 Ir < 0.05 Br < 0.1	Ge	< 0.1	Re	< 0.05	
Br < 0.1 Pt < 0.05 Rb < 0.05	As	< 0.1	Os	< 0.05	
Rb < 0.05 Au < 0.1 Sr < 0.05	Se	< 0.1	lr	< 0.05	
Sr < 0.05 Hq < 0.5 Y 0.09 Tl < 0.1	Br	< 0.1	Pt	< 0.05	
Y 0.09 TI < 0.1 Zr 1.2 Pb < 0.5 Nb < 0.1 Bi < 0.1	Rb	< 0.05	Au	< 0.1	
Y 0.09 TI < 0.1 Zr 1.2 Pb < 0.5 Nb < 0.1 Bi < 0.1	Sr	< 0.05	Hg	< 0.5	
Nb < 0.1 Bi < 0.1				< 0.1	
	Zr	1.2	Pb	< 0.5	
	Nb	< 0.1	Bi	< 0.1	
Mo 0.14 Th < 0.05	Мо	0.14	Th	< 0.05	
Ru < 0.1 U < 0.05	Ru	< 0.1	U	< 0.05	
Rh < 0.1	Rh	< 0.1			

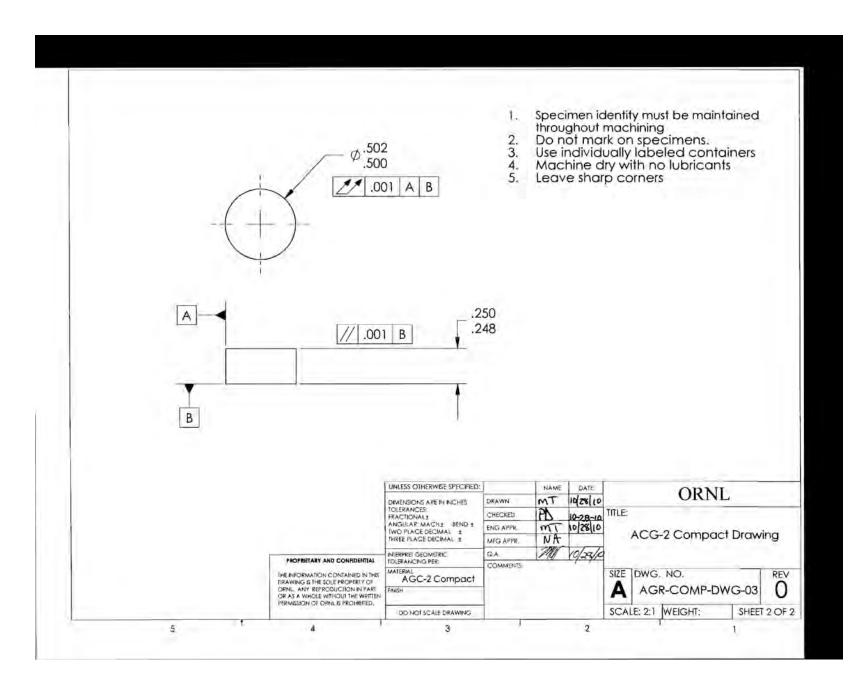
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Appendix A: Compact Machining Drawings

Prior to heat-treatment, carbonized cylinders were machined according to drawing AGR-COMP-DWG-03, "AGC Compact Drawing". Copies of these drawings are attached in this appendix.





Appendix B: Certificate of Conformance

This section contains the Certificate of Conformance for the A3-H08 compact lot. This is a record of the review by Quality Assurance personnel that specified requirements in INL SPC-1285, Rev. 1 have been met. All compacts selected for irradiation or impurity analysis met all dimensional and density specifications. Note that the specification only requires compacts used for irradiation meet these requirements, so the final determination of this inspection is that the product (compacts used for irradiation) conforms to the specified requirements for length, diameter, and density.

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Oak Ridge National Laboratory Advanced Gas Reactor Fuel Development and Qualification Program CERTIFICATE OF CONFORMANCE

2. PART		OT NUM	BER: INL	Blend F w	ith Hexic	on D_SD-1708 + rix Compacts fo			
	OF APPROVE								
*Part	Unique Part	QTY	INIT.	Date	*Part	Unique Part	QTY	INIT.	Date
Type	I.D. No.				Type	I.D. No.			-
MC	A3-H08-Z07	1	MIN	21/11/10					
MC	A3-H08-Z19	1	7/1/1/	Silulio					
MC	A3-H08-Z01	1	MIN	while					
				1//					
						-			
								_	
			-	-					
				1		-			
			-						
					-				
5. LIST	OF APPLICA	BLE NO	NCONFO	RMANCE	REPOR	T NUMBERS:	Not App	licable	
condition produced (Docume	ns documented d and tested in c	on Nonco compliance NL-AGR-	nformance e to the rec 01), its sub	Reports re quirements pordinate in	of the QA	referenced in Ite in Item 5, the li AP for the AGR ing procedures,	sted parts Program	have been at ORNL	
M. C. V	ance, AGR Qua	lity Repre	esentative,				-4	Date	2

Fuel Cycle and Isotopes Division, ORNL

^{*} MC indicates matrix-only compact